Washing effects on electrochemical performance and storage characteristics of LiNi$_{0.8}$Co$_{0.1}$Mn$_{0.1}$O$_2$ as cathode material for lithium-ion batteries

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HIGHLIGHTS

► The effect of water washing on fresh LiNi$_{0.8}$Co$_{0.1}$Mn$_{0.1}$O$_2$ cathode was firstly studied.
► Water washing improved the cycling performance and structural stability of material.
► Water washing resulted in a slight deleterious effect on the capacity.
► Water washing made material much less resistant to H$_2$O and CO$_2$ when storing in air.

GRAPHICAL ABSTRACT
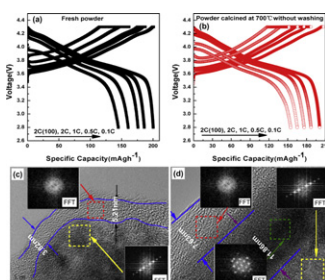
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ABSTRACT

The effect of water washing on LiNi$_{0.8}$Co$_{0.1}$Mn$_{0.1}$O$_2$ cathode is extensively studied with respect to the electrochemical properties, structural stabilities, and storage characteristics. Water washing can improve the cycling performance and structural stability of LiNi$_{0.8}$Co$_{0.1}$Mn$_{0.1}$O$_2$ material in electrolyte, although with a slight deleterious effect on the capacity. Storage tests of the 4.3 V charged electrodes at 90 °C after 30 days show that Ni, Co, Mn in fresh and washed samples are dissolved in electrolyte, but the amount of dissolved Ni^{4+} ions after washing decreases by 31.5% compared with the fresh cathode. Moreover, the fresh sample transforms into the spinel phase with a $Fd3m$ space group, whereas the washed sample remains as a layered hexagonal phase with an $Rm\overline{3}$ space group. FTIR spectroscopy, transmission electron microscope (TEM), X-ray diffraction (XRD) and electrochemical studies indicate that the washed materials become more easily attached upon exposure to air, accompanied by significant increase in cationic disorder and NiO-like cubic phase near surface.

1. Introduction

Layered composite Ni-rich materials are considered to be the most promising materials for hybrid electric vehicles, plug-in hybrid vehicles, and electric vehicles due to their large capacity, excellent rate capability, and low cost [1–3]. However, the problems of rapidly moisture up-taking and Li$_2$CO$_3$/LiOH impurities formed on surface upon exposure to air have hindered the practical applications. And the storage property of Ni-rich materials, which is directly related to the material production and electrode fabrication, has attracted considerable attention [4–13]. The formation of Li$_2$CO$_3$/LiOH on surface was thought to be the main source of poor storage property of Ni-rich materials [7–12]. Worse, LiOH reacted with LiPF$_6$ in the electrolyte to form HF. At the charged state, HF directly dissolves the transition ions in the cathode, leading to gas evolution and phase transformation from the layered to the spinel. The formation of Li$_2$CO$_3$/LiOH may be caused by the reactions between Ni-rich materials and the atmosphere. These reactions should include surface physical/chemical adsorption and possibly direct redox reactions between the bulk material and CO$_2$/H$_2$O in air. Zhuang et al. [7] found that air exposure of LiNi$_{0.8}$Co$_{0.15}$Al$_{0.05}$O$_2$ cathodes produced a 10 nm thin layer of Li$_2$CO$_3$ which produced

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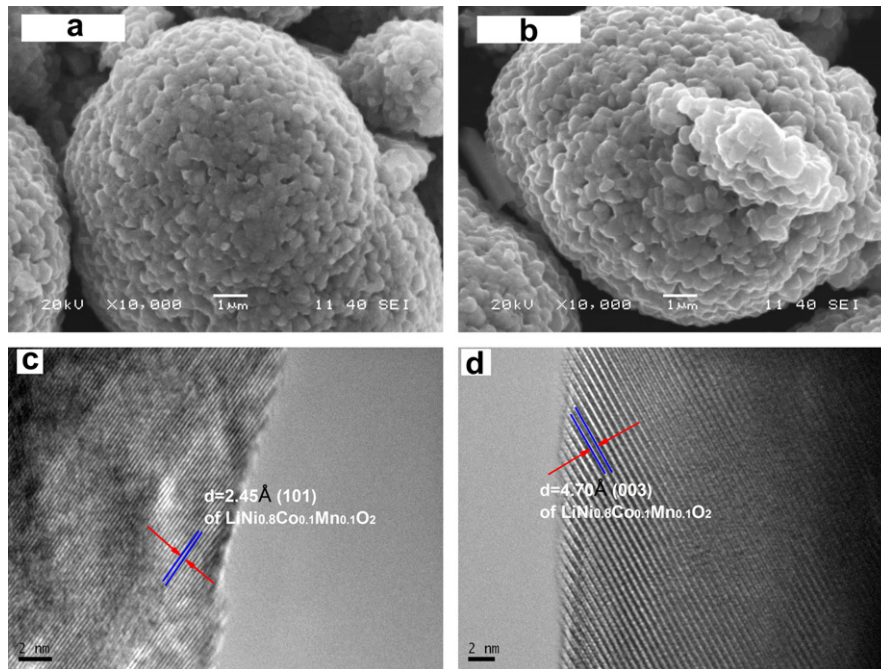 E-mail address: xxhui881118@126.com (Z. Wang).

Fig. 1. SEM and TEM images of F1 (a, c) and W2 (c, d).

severe deleterious effects on the capacity of cathodes in lithium ion battery. They thought that Li_2CO_3 had been formed by reaction of atmospheric carbon dioxide with lithium oxide residue in the powder and/or by reaction with Li from the $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ active material. K. Shizuka [11] reported there were two steps in the carbonation reactions; the first occurred with the excess Li components, and the second with $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ itself. Liu et al. [12] reported that during storage in air, a reduction of Ni^{3+} to Ni^{2+} on surface could produce active oxygen species, which then reacted with trace H_2O and CO_2 in air, and further combined with Li^+ to form $LiOH/Li_2CO_3$. Obviously, the formation of $LiOH/Li_2CO_3$ on the material surface can be ascribed to these two processes which take place at the same time during storage.

Another problem with Ni-rich materials is related to structural instability above 60 °C. Ni and active metal ions dissolving into the electrolytes during storage above 60 °C resulted in a partial phase

transformation to spinel phase [6,14,15]. This decreases the safety performances.

An effective way to slow down the rate of these side reactions is to create an artificial physical barrier. The protective layer must be thick enough to avoid contacting with the H_2O/CO_2 in air or electrolyte, and also the thickness must be kept within a few nanometers in order to enable Li^+ diffusion and to keep a reasonable electronic conductivity. MgO, SiO_2, AlF_3, $Ni_3(PO_4)_2$, $Co_3(PO_4)_2$, $AlPO_4$ have been intensively investigated [16–21]. Compared with bare samples, all these coated materials exhibited good cycle stability and improved thermal stability.

As described by Z.H. Chen [22], most surface coatings involve mechanically mixing of materials with precursors and heat treatment of mixtures at elevated temperatures to form the desired coating material on surface. With this type of coating technique, mixing of the materials with precursor material is very rough; some areas are heavily coated, while other areas are only barely coated. And areas barely coated may be regarded as simple washing when coating process is accomplished using water as solvent. In addition, simple washing can remove the excess Li on surface. Owing to the excess lithium present in the form of $Li_2O/LiOH$ on fresh powder would also account for the very rapid appearance of $LiOH/Li_2CO_3$ on surface of fresh sample exposure to air. Surface coating is an

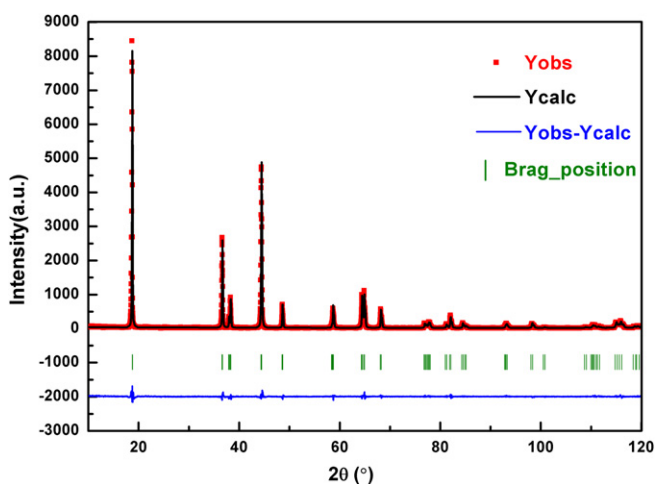**Fig. 2.** Rietveld analysis of the XRD pattern of $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$.

Table 1

Rietveld analysis results of sample F1, F2, W1, W2 and sample F1 and W2 after storage in air for 30 days.

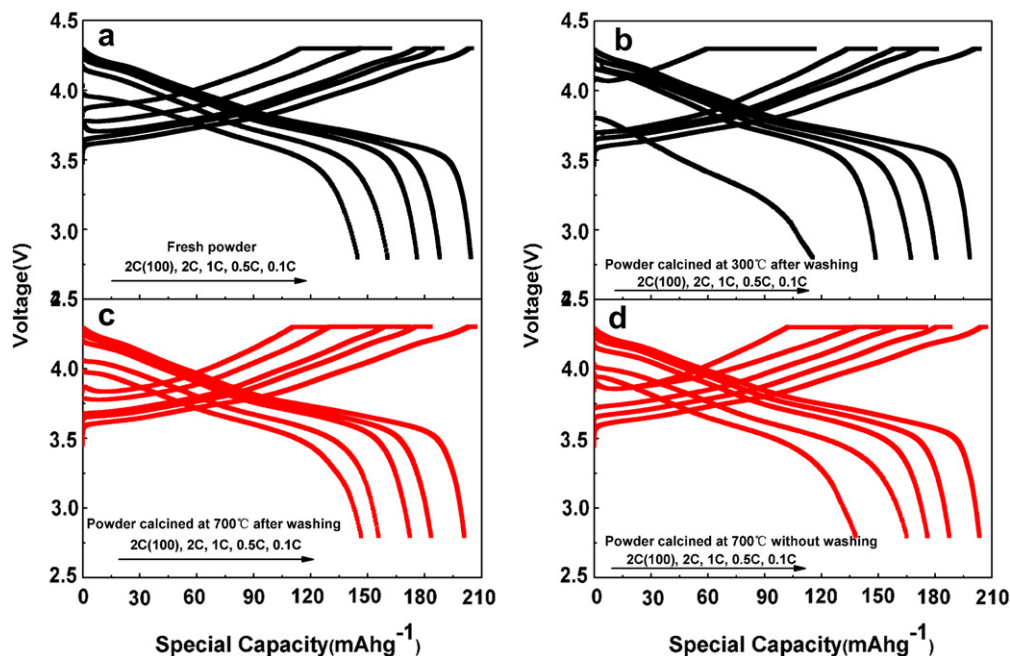| Sample	a (Å)	c (Å)	c/a	Ni in Li site	Rp/Rwp/Rexp
F1	2.87099	14.19679	4.9449	0.024	7.41/9.93/7.99
F2	2.87142	14.19773	4.9445	0.024	7.72/10.25/8.77
W1	2.87095	14.19583	4.9448	0.024	8.28/10.78/9.13
W2	2.87103	14.19638	4.9446	0.024	7.83/10.18/8.92
F1 after storage in air for 30 days	2.88869	14.22189	4.9233	0.038	9.11/12.09/9.55
W2 after storage in air for 30 days	2.89587	14.23204	4.9146	0.053	10.46/13.34/10.87

Fig. 3. The initial capacities at various C rates and cycling life at 2 C rate of sample F1 (a), W1 (b), W2 (c) and F2 (d).

effective way to improve the property of $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$, and the role of surface coating is often extensively study between pristine and coated sample. However, there is no comparatively study between washed sample and pristine sample, or coated sample. It's very imperative and interesting to study whether zero coating, namely the simple washing, has effect on material's properties. Therefore, in this paper, we have studied the effect of washing on structural and electrochemical properties, and storage characteristics of $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ powder.

2. Experimental

$LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ powder was prepared by mixing co-precipitated $Ni_{0.8}Co_{0.1}Mn_{0.1}(OH)_2$ and $LiOH·H_2O$ and firing at 480 and 750 °C for 5 and 15 h, respectively, at a molar ratio of 1:1.05. After sieving, powders with an average particle size of 7 μm were marked as F1 and used for further experiments. The washing process was simulated by adding 20 g F1 powder to 40 mL purified water under constant stirring with a magnetic stirrer for 20 min. As reported, almost all coating process accomplished followed by heat treatment at 300–800 °C. So in order to simulate the coating process, the powders fired at 300 or 700 °C after recovered by filtration, and marked as W1 and W2, respectively. The powder re-fired at 700 °C without washing was marked as F2. The storage process was carried out at room temperature by storing the sample in air for 7 days and 30 days. After storage, the change in the pH of the powders immersed in water was monitored by adding 5 g powder to 50 mL purified water under constant stirring. Since $Li_2CO_3/LiOH$ were dissolved in water, and most of the Li sources were believed to originate from $LiOH/Li_2CO_3$ on surface, the total weight of the Li sources in filtrate could be measured using a titration method.

Electrochemical charge–discharge tests were performed using the cathode with a mixture of 80 wt.% cathode material, 10 wt.% Super P carbon black and 10 wt.% polyvinylidene fluoride (PVDF). The electrolyte was 1.0 M $LiPF_6/EC$ + DMC + EMC (1:1:1, volume ratio). Preliminary cell tests were done using 2025 coin-type cell

adopting Li metal as an anode with a voltage window of 2.8–4.3 V (versus Li/Li^+) at room temperature.

The amount of Ni, Co and Mn dissolved from the fresh and washed $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ cathode material was measured by inductively coupled plasma (ICP). Powder X-ray diffraction (XRD, Rint-2000, Rigaku) measurements using Cu Kα radiation were used to identify the crystalline phase of powder. The SEM images of particles were observed with scanning electron microscopy (SEM, Sirion 200). Transmission electron microscopy (TEM, Tecnai G12) was carried out using a Philips CM200 microscope with energy dispersive X-ray analysis (EDX). Fourier transform infrared (FT-IR) spectra were recorded by a Nicolet AVATAR 360 FTIR spectrometer.

3. Results and discussion

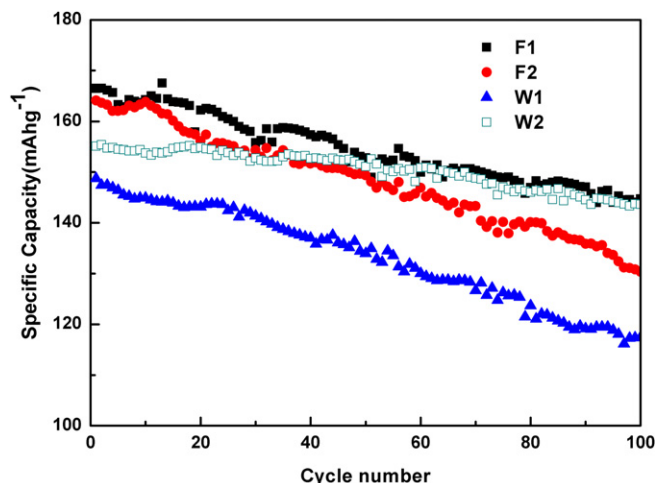Fig. 1 shows a comparison of the SEM micrographs of sample F1 and W2, there are no obvious changes in morphology after

Fig. 4. The cycle performance at 2 C rate of sample F1 (a), W1 (b), W2 (c) and F2 (d).
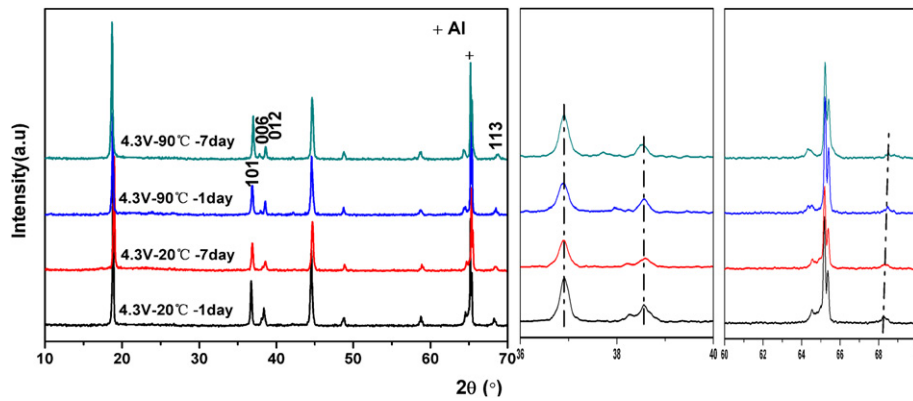
Fig. 5. XRD patterns of sample F1 after charging at 4.3 V at various storage conditions.
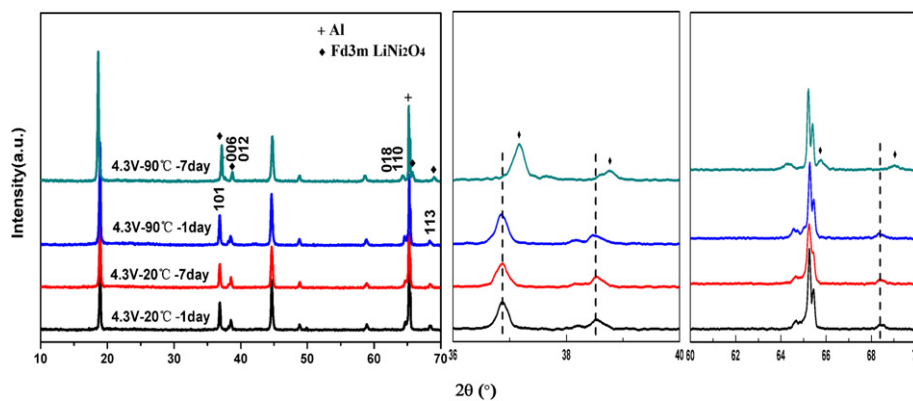

Fig. 6. XRD patterns of sample W2 after charging at 4.3 V at various storage conditions.

washing. The TEM image of sample W2 in Fig. 1(d) shows good crystallinity at the edges of the grains and no evidence for a surface film, as opposed to that of sample F1, which contains fuzzy surface. According to the research results of G.V. Zhuang [6] and K. Shizuka [7], the excess lithium is not in the crystal but on the surface, it is reasonable to think the fuzzy surface is $Li_2O/LiOH$ layer. This is confirmed by the pH value of sample F1 rising to 12.4 (listed in Fig. 8) immediately when immersed in water.

The amount of Ni in the Li sites of four samples was estimated by a Rietveld analysis of the XRD patterns in Fig. 2. The lattice constants a and c calculated from the Rietveld analysis are shown Table 1. A few researchers [9,12,23] had found the degree of cationic disorder in the layered structure increased after contaction with H_2O/CO_2 in air for $LiNiO_2$-based materials. But the data in Table 1 indicate washing will not influence the lattice constants, as well as the Ni amounts in Li sites. This is because the time of immersed in water is too short to observe the changes in chemical states of the powders.

As shown in Figs. 3 and 4, the initial capacities and cycling life of sample F1, F2, W1 and W2 are compared in order to determine the effects of washing and heat-treatment on the electrochemical properties. As observed in Figs. 3 and 4, the washed materials show lower specific capacity at low C rate than fresh materials. The extraction in water leads to a considerable chemical delithiation accompanied by the loss of oxygen from the crystal lattice of $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ and the defective NiO octahedra cannot release electrons whereby the amount of Li^+ which can be deintercalated during the electrochemical delithiation is reduced [5]. And the first discharge capacity at 1 C and 2 C of sample W1 was 167.3 and

148.7 mAh g^{-1}, which is much lower than that of other sample, and the capacity retention of this samples after 100 cycles at 2 C rate was only 115.4 mAh g^{-1}, which is 13.6% lower than that of sample F1. However, the cycling performance at 2 C rate of sample W2 is most outstanding. According to the report of Abraham et al. [6] and Liu et al. [12], NiO-like thin layer near surface region will create due to the reaction of $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ with H_2O during the washing process. When the temperature of heat-treatment is low, the NiO-layer can't be re-oxidized and it's electrochemically inactive to lithium intercalation/deintercalation, which is believed to be responsible for the performance loss of sample W1. As shown, after heat treatment at 700 °C in O_2 flow, the cycling performance at 2 C rate is improved because NiO-layer is re-oxidized and reacts with unremoved synthetic residue $Li_2O/LiOH$. Moreover, most of the synthetic residue $Li_2O/LiOH$ which is believed to produce HF will be removed and the structure will be enhanced (represented in Fig. 5). This is cause of the capacity retention of sample W2 is much higher than sample F1. The sample F2 shows a rapid capacity drop to

Table 2
Dissolved metal ion contents in $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ after storage at 90 °C (cathodes were charged to 4.3 V). Units are ppm.

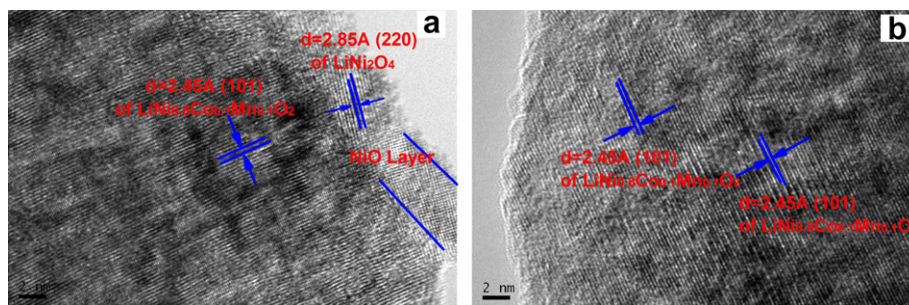| Sample		Ni	Co	Mn
F1	7 days	205	27.8	6.1
	30 days	1152	123.5	18.6
W2	7 days	105	20.4	4.3
	30 days	789	79.3	12.1
F2	7 days	293	37.9	9.7
	30 days	1466	151.4	33.2

Fig. 7. TEM analysis of (a) sample F1 and (b) sample W2 after storage at 90 °C for 7 days at charged state.

130.9 mAh g^{-1} and a 6.5% deteriorated capacity after 100 cycles at 2 C compared with sample F1. N. Mijung et al. [23] reported a very small oxygen-rich stoichiometry in the LiNi$_{1/3}$Co$_{1/3}$Mn$_{1/3}$O$_2$ produced during annealing and it resulted in capacity deterioration, but this phenomenon is not found in our TG-DTA experiments. It's not clear about this severe capacity loss at this moment. More sensitive methods may be necessary to study the effect of heat treatment.

The purpose of our research is to study the effect of water in the coating process using water as solvent. Similarly to study the role of surface coating, sample F1 and W2 are chosen for further research. The structural stability of sample F1 and W2 after charging at 4.3 V is investigated in Figs. 5 and 6. At 20 °C, regardless of the storage time in electrolyte, no noticeable structural changes are observed in both sample F1 and W2. But the XRD patterns of two samples reveal different phenomenon after storing at 90 °C. After stored at 90 °C for 7 days, (006)/(012) peaks of fresh sample merging into the single ones, and the (101), (113) and (006)/(012) peaks shifting to higher angle, indicating that the layered phase is transforming to the spinel LiNi$_2$O$_4$ phase ($Fd3m$). However, sample W2 stored at 90 °C for 7 days does not show any obvious changes, implying that the original layered structure with an $R\bar{m}3$ space group is sustained.

Table 2 shows the concentration of Ni, Mn and Co cations detected in the electrolyte for charged sample F1 and W2 stored at 90 °C for 7 days and 30 days. The concentration of Ni and Co cations are considerably higher than that of Mn cations at each condition, this is because the oxidant Ni^{4+} and Co^{4+} ions are not stable as

independent ions. Structural stability of the material may be worsened due to the possible electrolyte oxidation at the electrode/electrolyte interface and the increasing dissolution of the active material into the electrolyte at high temperature. Mn^{4+} ions in LiNi$_{0.8}$Co$_{0.1}$Mn$_{0.1}$O$_2$ material did not participate in redox reaction, the dissolved amounts of Mn ions in all cathodes are negligible. As shown in Table 2, the dissolved metal contents of sample W2 are much lower, it is believed that most of the synthetic residues Li$_2$O/LiOH on surface are washed and the amount of HF produced by Li$_2$O/LiOH decreases. Meanwhile, sample F2 shows the highest dissolution of transition metal ions. This indicates that heat-treatment may accelerate the formation of Li$_2$O/LiOH and the severe capacity loss of sample F2 may ascribe to more Li$_2$O/LiOH on surface.

TEM analysis of the charged samples F1 and W2 after storage at 90 °C for 7 days is carried out, as shown in Fig. 7. It is evident that the near surface region of sample W2 is distinctly different from the bulk. In Fig. 7(a) a lattice fringe of the (220) plane of the spinel LiNi$_2$O$_4$ phase corresponding to 2.85 Å is observed, as well as rock salt (NaCl-type) crystal NiO layer. However, sample W2 does not show such phase transition to a spinel phase, as seen in Fig. 7(b). These all confirm the XRD results.

Fig. 8 compares the pH value changes in sample F1, F2 and W2, as a function of time. As soon as the sample F1 and F2 are placed into the water, the pH values increases to 12.4 and 12.7 in a few minutes and stabilizes at those values, respectively. However, sample W2 shows the lowest value of pH at 11.8, indicative of the
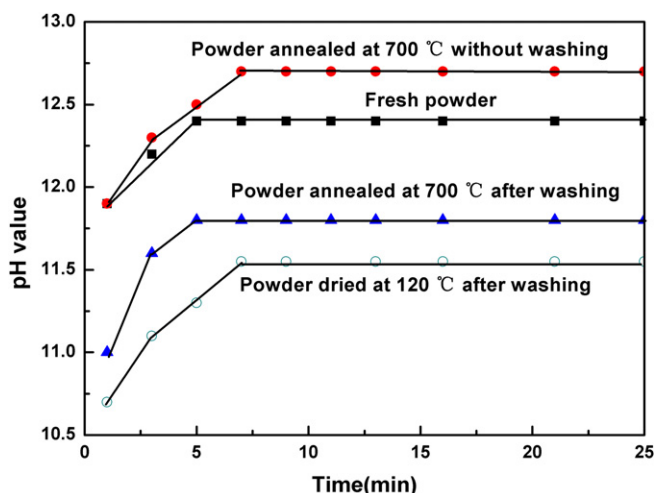

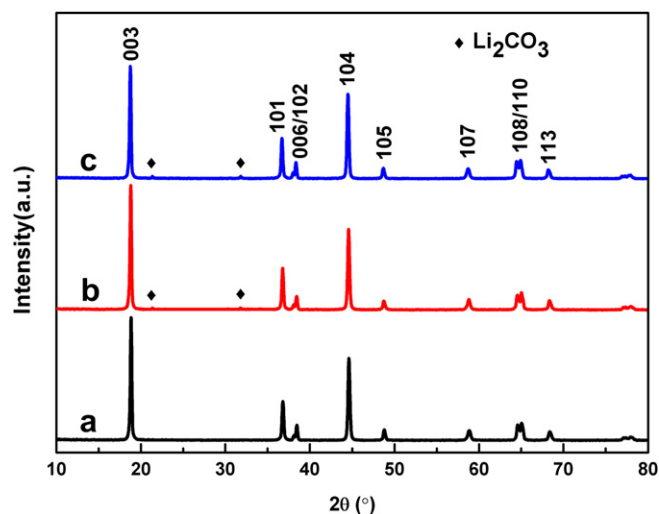**Fig. 8.** pH value changes as a function of time in different powders.

Fig. 9. XRD patterns of sample F1 before (a) and after storage (b) and sample F2 after storage (c) for 30 days.
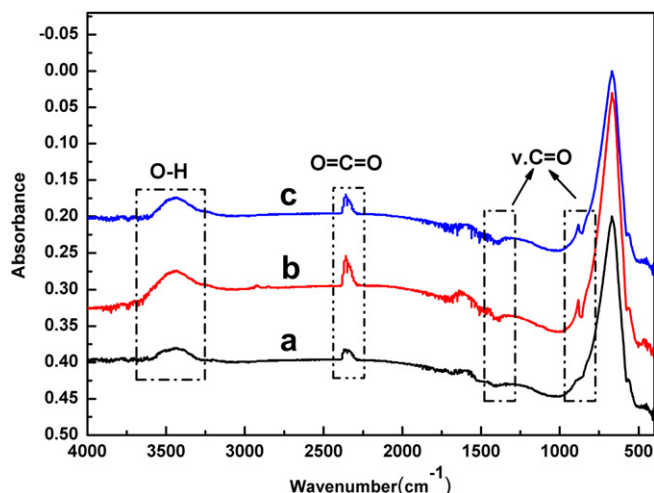
Fig. 10. FT-IR spectrum of sample F1 before (a) and after storage (b) and sample W2 after storage (c) for 30 days.

removal of lithium impurities on surface during washing process. Both of the phenomenons that sample F2 shows the highest pH and the pH value of powder dried at 120 °C is lower than that of sample W2 suggest that heat-treatment accelerates the formation of Li_2O/LiOH on surface. More $LiOH$/Li_2CO_3 forms during electrodes manufacture process on surface of sample F2 and particle isolation of active material is proposed as the mechanism for capacity loss in initial cycles relative to fresh electrode. The bad cycling performance is due to more HF produced from reaction between electrolyte and $LiOH$/Li_2CO_3.

From the above discussion, we can know water washing can improve the cycling performance and structural stability of the $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ material storing in electrolyte, although the capacity loss associated with water washing cannot be neglected.

However, a problem for scale-up with the $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ material is rapid moisture uptake upon exposure to air. Of course, it is worth studying the storage performance in air.

Fig. 9 shows XRD patterns of sample F1 before and after storage and sample W2 after storage. Two weak peaks located at 21.3° and 31.8° which are identified as Li_2CO_3 phase appear in the XRD patterns of the stored samples. According to the refinement results in Table 1, both the lattice parameters a and c of the stored sample became larger than that of fresh sample. Moreover, the degree of cationic disorder in sample W2 is more severe, and more nickel ions with inactive cubic rock salt phase will appear in this sample. Specific amount of Li_2CO_3/LiOH can be calculated in both of stale $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ materials in following detection.

The FT-IR results (Fig. 10) give more evidence of the surface changes on $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ during the storage. Compared with sample F1, the FT-IR spectrum of the stale materials shows significantly increased peaks of LiOH and Li_2CO_3. The band at 3650–3200 cm^{-1} is assigned to the O–H stretching vibration from LiOH, and two peaks at 1450 and 870 cm^{-1}, which are assigned to the CO_3 vibration from Li_2CO_3. The peaks of these bands for the sample W2 are more prominent than those of the sample F1 after storing in air. This means that the amount of LiOH/ Li_2CO_3 on the surface of sample F1 is less than that of sample W2 after exposure to air. Washing leads to the easy removal of LiOH/ Li_2O on fresh particles, but makes it much less resistant to H_2O/ CO_2. This is very different from the report of J. Kim et al. [8]. It's very interesting and further research of the mechanism is in progress in our lab.

In order to verify the FT-IR results, the differences of stale materials were identified by SEM and TEM. For the stale samples, the particle morphology has obviously changed. The SEM images (Fig. 11 (a) and (b)) show a layer of transparent substance covering the particles, while sample F1 (Fig. 1(a)) looks very smooth. The differences in crystal structure between the bulk and near-surface regions are evident in Fig. 11(c) and (d), which show HRTEM
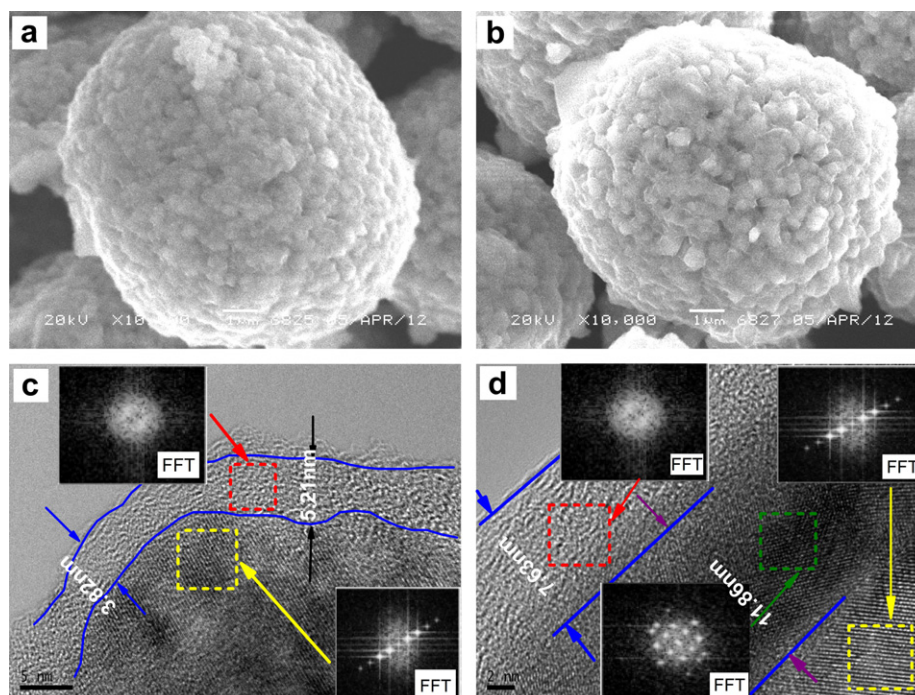

Fig. 11. SEM and HREM images of sample F1 (a, c) and W2 (b, d) after storing in air for 30 days.

Table 3
The LiOH and Li_2CO_3 content in fresh powder and powder annealed at 700 °C after washing after storage in air for 7 and 30 days. Units are ppm.

	LiOH (7 days)	Li_2CO_3 (7 days)	LiOH (30 days)	Li_2CO_3 (30 days)
F1 after storage in air	412	1287	1222	4506
W2 after storage in air	623	1899	2291	7069

images and the corresponding fast Fourier transform (FFT) patterns. As shown in Fig. 11(c) and (d), the surface film visible on the particle contains amorphous layers and elemental analysis by EDX (the data are omitted) shows the coating layer is composed primarily of carbon and oxygen, which is consistent with its identification as lithium carbonate by FT-IR and earlier reports (Zhuang et al. [7] and J. Eom et al. [15]). The thickness of amorphous layers are 3.82–5.21 and 7.63 nm for sample F1 and W2 after storing in air, respectively. In addition, it is evident that there exists a transition layer between amorphous layer and the bulk for sample W2. And the transition layer is distinctly different from them and is confirmed by FFT patterns. It is concluded that the transition layer is NiO layer accompanied by the loss of the oxygen and Li when storing in air (Liu et al. [9]). More Ni in Li site indicates more nickel ions with inactive cubic rock salt phase appears, we can know that sample W2 will produce more NiO phase from the Rietveld analysis for stored samples. Hence, it is reasonable to suggest water washed $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ becomes more easily attached and produce more NiO-like cubic phase when storing in air. Meanwhile, NiO layer is too thin to be identified in Fig. 11(c) and we can infer that most $Li_2CO_3/LiOH$ come from synthetic residue $Li_2O/LiOH$ for sample F1 after storing in air for 30 days. This Worse performance is expectable because of NiO layer and severe cation mixing for sample W2.

In order to measure the actual amount of $LiOH/Li_2CO_3$ on $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ surface, the stale powders are soaked in water

and the actual amount of LiOH and Li_2CO_3 are measured using titration method. As shown in Table 3, this is in good agreement with the FT-IR and TEM results.

The first charge–discharge curves under different current density and cycling performance of fresh and washed powders after storage in air for 7 and 30 days are shown in Fig. 12. All the cathodes show a large capacity decrease. Washed powder shows drastic capacity fading especially the cycling performance at 2 C rate. For instance, the stored samples F1 and W2 exhibit discharge capacities of 102.0 and 64.7 mAh g^{-1} after 100 cycles at 2 C, respectively. This is coincident with the trends of the amount of $LiOH/Li_2CO_3$ on surface of stale materials and the degree of cationic disorder. It is reasonable to think that the deterioration of $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ during storage is attributed to the formation of $LiOH/Li_2CO_3$ and the structural changes of the surface.

4. Conclusions

The cycling performance and structural stability of $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ material after storing in electrolyte were enhanced by water washing, although it produced a slight deleterious effect on the capacity of $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ material. ICP, TEM and EDX analysis indicated that the improved structural stability was ascribed to the removal of synthetic residue $Li_2O/LiOH$. However, water washing made $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ material much less resistant to H_2O and CO_2 when storing in air. The amounts of $LiOH/Li_2CO_3$ on washed sample were almost 2 times that of fresh sample after storing in air for 30 days, as well as the degree of cationic disorder. Accordingly, the electrochemical performance of the washed material after storing in air becomes poorer. This property will be valuable for its practical use. Because more and more studies aimed at enhancing electrochemical performance and storage characteristics by the coating are currently underway, when lots of bared areas exist, they remain weak sites for the attack of non-aqueous electrolytes and CO_2/H_2O and the protection will not maximized.
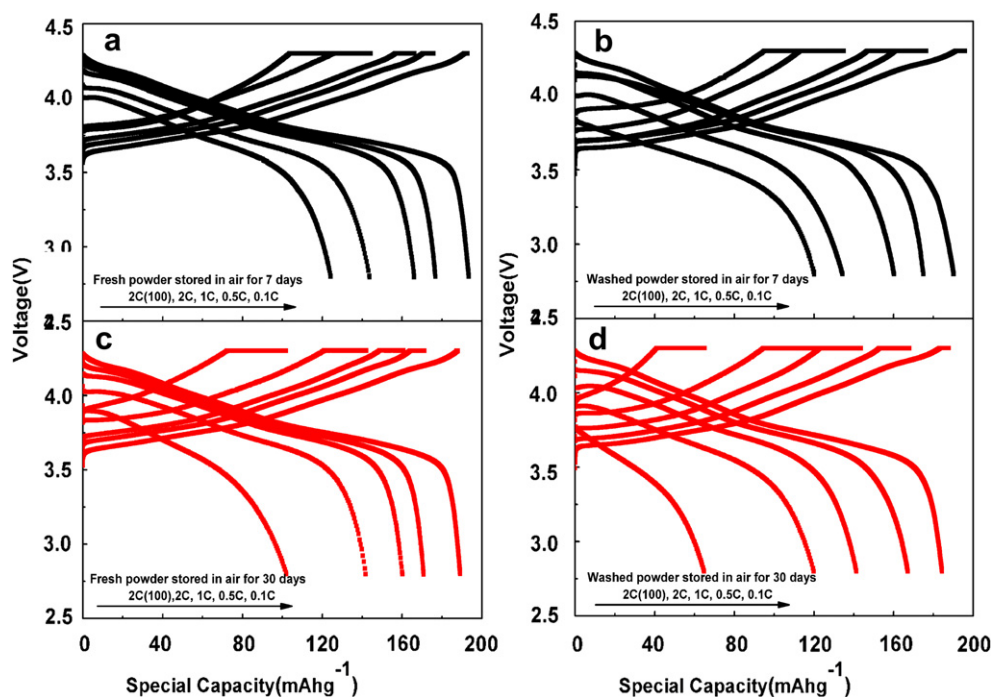

Fig. 12. The first charge–discharge curves under different current density and cycling performance of fresh and washed powders after storage in air for 7 and 30 days.

Acknowledgments

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